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Mineralogical studies and optimization of tabling parameters of low grade chromite ore by box behnken design of experiments

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Abstract

Chromite ore is geologically associated with gangue materials which have lower density compared to pure chromite ($FeO.Cr_2O_3$). Gravity separation techniques are very suitable for beneficiation of low grade chromite ore. In the present study mineralogical analysis of the sample has been carried out by using Wavelength dispersive x-ray fluorescence (WDXRF), X-ray powder diffraction (XRD), Scanning electron microscopy (SEM) and Energy dispersive X-ray (EDX). The beneficiation of low grade (Cr_2O_3% 26.88) chromite ore has been investigated using wilfley table. Optimization has been carried out using box-behnken statistical design (BBD). The experimental parameters selected are: tilt angle, water flow rate and pre-heat treatment of the chromite ore. It is observed that percentage recovery of Cr_2O_3 depends upon tilt angle, water flow rate and pre-heat treatment of chromite ore. Pre-heat treatment of chromite ore plays a vital role in gravity separation. The optimized tabling conditions suggested by BBD are 8° tilt angle, 4 $L \cdot min^{-1}$ water flow rate and temperature for pre-heat treatment of raw chromite ore at 443.72°C. After tabling under optimized conditions, chromite concentrate contained 1.91 Cr/Fe ratio, 46.02% of Cr_2O_3 with 65.95% recovery.

1. Introduction

Chromite ore is the primary source of chromium. India has large reserves of chromite which is about 344.016 million tons. Orissa alone accounts for 330.714 million tons and most of it is located in Sukinda region [1]. Chromite has various applications in steel industry, automobile industry, rails, refractory industry (owing to its high corrosion resistance and high melting property), cement, non-ferrous alloy industry, chemical industry as chrome chemical and chromium plating solution, etc.

Chromium exists as chromium spinel in chromite ores along with magnesium, aluminium and iron in different proportions depending upon the deposits. Chromite is chemically represented as $FeO.Cr_2O_3$, where Fe is replaceable by Mg and Cr by Al or Fe [2]. Chromite ore can be classified into three categories: high grade chromite ore containing 50% Cr_2O_3, sub grade chromite ore containing 35% to 40% Cr_2O_3 and low grade chromite ore containing 20% to 25% Cr_2O_3. High grade chromite ore is directly transferred to the market for ferroalloy production. Beneficiation of ore is required in case of sub grade and low grade chromite ores to make them marketable.

Comminution process is used for liberation of gangue particles like serpentine, olivine, quartz, etc. from chromite ore. Two basic comminution processes are crushing and grinding. Different crushing equipments like jaw crusher, cone crusher and roll crusher are used for crushing whereas ball mill and roll mill are generally used for grinding purposes. Different beneficiation processes have been used to increase the concentration of low grade chromite ore. The processes commonly used are gravity separation, froth-flotation, electrostatic separation, magnetic separation and selective flocculation. Beneficiation of chromite ore has been done by many researchers to improve the Cr/Fe ratio using different beneficiation techniques [3-7].

G. P. Gallios et al. have studied the behavior of the chromite ore and separation of chromite from serpentine by using flotation process. Experimental result indicates that sodium oleat as a collector in alkaline medium plays very important role for the flotation process of low grade chromite ore [8]. Flotation is a very good technique and gives significant results for the up gradation of chromite ore. The problem arises since different chemicals are used which are not environment friendly. Apart from that this process completely depends upon the nature of the gangue minerals. Chromite ore containing serpentine as gangue minerals is very difficult to separate by this process due to the similar surface phenomena of both minerals [9]. The choice of collector is important in this process, which is controlled by pH of the investigated media. For example anionic collectors below pH 6 cannot be used in this process. In this process dimerization, miscellization, and precipitation like phenomenons are involved [4]. This process is a good alternative method for chromite ore beneficiation.

Selective flocculation process is also useful for beneficiation of low grade chromite fines below 150 μm. The basic principal of this technique is adsorption of different flocculants on the selective mineral particles and helping them settle down faster than the rest of the minerals present in the ore body. The impure minerals are present in the solution as suspension after the adsorption of flocculant. Starch and different polyacrylamides have good affinity towards chromite than serpentine above pH 11. This process is applicable for chromite recovery when lower amount of serpentine is present in the ore body as gangue materials [10-11]. The problem arises when higher amount of serpentine is associated with ore body, which prevents the separation of chromite from the gangue materials.

Heavy media separation (HMS) technique is commonly applied for separation of sand, coal and high density minerals from their ore like lithium, manganese, chromium bearing ores. The particle size optimization of the feed sample is important before it is subjected to HMS. Mixture of bromofrom and carbon tetrachloride give densities in the range 1.58 $g \cdot cm^{-3}$ to 2.89 $g \cdot cm^{-3}$ which is mainly used as heavy media for industrial mineral processing. Alloys like ferrosilicon (S.G. 6.7 to 6.9) are used as a heavy media for the upgradation of low grade chromite ore of the Ingassana hills [12]. Recycling of media is crucial in this process because it is very expensive. The wastage of heavy media increases the operational cost.

High intensity induced roll magnetic separator is also used for the chromite ore up gradation process. Nevzat Aslan *et al* used multi gravity separator and high intensity induced roll magnetic roll separator for chromite beneficiation and optimization of process parameters has been carried out by taguchi method. It is observed that the grade and recovery improve up to 47.74% and 73.31% from 42.38% and 65.34%; respectively [4]. The effect of feed particle size, drum speed and washing water on multi gravity separator are vital for chromite recovery [13]. By using simple magnetic separator Cr/Fe can be improved up to 2.42 from 1.49 [14]. S. K. Tripathy *et al* also have investigated the chromite ore beneficiation using four different types of magnetic separators. The investigation report shows that wet high-intensity magnetic separator and induced roll magnetic separator gives better product quality with chromium oxide recovery. It is also observed that to obtain better product quality and recovery two-stage operation are required at higher and lower magnetic field intensities [15]. But the application of this process is suitable for ferruginous chromite ores, otherwise it is a very costly process due to the expensive electrical energy involved in the process. The recovery percentage of chromium oxide is also not significant in magnetic separation process. The reason behind that may be due to the chromite particles being coated with magnetic particles and during magnetic separation it goes to the magnetic fraction which is treated as waste product in that case.

Nowadays gravity separation technique is widely used for chromite ore beneficiation. Gravity separation is mainly carried out by wilfley table, spiral gravity separator, heavy-liquid separation and jigging. Gravity separation techniques are very effective for chromite ore beneficiation as reported by many researchers [16-21]. In 2012, A. Seifelnasr et al. upgraded low grade chromite ore containing 20% to 35% Cr_2O_3 using tabling. Percentage chromium oxide is improved upto 49% with 63% of recovery while raw chromite ore contained only 34.2% Cr_2O_3. Author concluded that separating the materials into size fraction without desliming is more effective than desliming the materials [4]. Sunil Kumar Tripathy *et al.* used wilfley table for gravity separation of lean grade chromite which contained 40.8% Cr_2O_3 with Cr/Fe ratio 1.7. After separation concentrate fraction contained 48.9% Cr_2O_3 with Cr/Fe ratio 2.31 [5]. In 2020 A. K. Das *et al*. also investigated the beneficiation behavior of low grade chromite ore for ferrochrome production by using wet shaking table and wet high-intensity magnetic separator (WHIMS). It has been observed that low grade chromite ore can upgraded up to 42.5% from 16% by using wet shaking table and WHIMS [22]. From the above discussions, it can be seen that gravity separation techniques have some advantages compared to other beneficiation techniques especially in case of chromite ore. Gravity separation via wilfley table has advantages like, high selectivity with high upgradation ratio if it is handled properly, very easy to operate, low operational cost, lesser number of trials are necessary to achieve the desired separation and easy adjustments can be made to the different parameters [23].

However, in present scenario the availability of high grade chromite ore is declining due to its huge demand and consumption in the alloy steel industry. Besides this, huge chromite fines are generated during chromite mining. Discarding these fines or using the fines in less important areas is equivalent to the loss/inappropriate uses of chromium and other valuable metals present in the fines. The detailed optimization study of low grade chromite ore is limited and not much references are available. In the present investigation an attempt has been made to provide a noble technique of heating followed by air quenching to generate a lot of physical defects for easy subsequent processing. This is the main feature of the study to see the effect on beneficiation of chromite ore by initiating cracks & fissures for liberation of valuable minerals. The objective of the present study is to utilize the lean grade ore by beneficiation and generation of cracks & fissures in the raw materials leading to probable increase of reducibility of upgraded materials. In the present experiment, characterization studies of the raw materials have been carried out by using WDXRF, XRD and SEM-EDX. Laboratory scale wilfley table has been used for beneficiation of low grade chromite. Three different parameters have been selected for the experiment. Effects of tilt angle, water flow rate and pre-heat treatment of chromite ore before charging on wilfley table have been investigated. The optimization of parameters has been carried out using BBD.

2. Experimental

2.1 Materials and method

Chromite ore has been crushed and ground. The crushed ore has been sieved and chromite ore fines below 75 µm have been selected. The selected sample have then been mixed homogeneously for further analysis and testing. After mineralogical studies, the samples are divided into three parts and subjected to pre-heat treatment for 30 min in an oxidizing atmosphere at 28°C, 514°C and 1000°C respectively in raising hearth furnace. The choice of temperatures has been carried out with the help of thermogravimetry and differential thermal analysis (TG-DTA). Three different samples are homogeneously mixed and subjected to gravity separation using the set of combination of parameters suggested by the BBD model. In that experiment laboratory scale wilfley table of 37 cm × 100 cm is used for the enrichment of the low grade chromite ore. The investigating parameters are pre-heating of raw chromite ore, tilt angle and water flow rate. The other parameters which are fixed for every experiment are given below:

Feed rate: 100 g/min where the solid liquid ratio is 1:6

Particle size fraction of feed: 100% below 75 µm

Vibration rate: 250 rpm

Riffles height: not fixed but approximately 2 mm

Distance between riffles: not fixed but approximately 14 mm

For all the experiments the position of collection containers has been fixed for concentrate, middling and tailing products. After each experiment, samples are collected from their fixed positions for further analysis. The overall process flow sheet of experimental setup is shown in Figure 1.

2.2 Mineralogical analysis of chromite ore

2.2.1 WD-XRF analysis of chromite ore

Low grade chromite ore is collected from Sukinda mines with the assistance of Tata Steel. The samples are than crushed & ground to fine particle size with the help of jaw crusher, roll crusher and pulverizes followed by sizing using a BSS standard sieve. The fine particles (below 75 µm) of chromite ore are analyzed using Wavelength dispersive x-ray fluorescence (WDXRF; PAN analytical), result is shown in Table 1. WD-XRF analysis shows chromite sample contains 26.88% Cr_2O_3, 23.72% Fe, 21.07% Al_2O_3, 5.1% MgO, 0.92% SiO_2.

2.2.2 XRD analysis of chromite ore

XRD pattern of the raw ore samples is given in Figure 2. The figure shows that the major peak is berezovskite $(Mg,Fe)(Al,Cr)_2O_4$ along with minor peaks of hematite (Fe_2O_3), aluminium hydroxide $Al(OH)_3$ and quartz. Phase analysis is performed by X-ray diffractometers (XRD; Rigaku Ultima III) using Cu K_α which is shown in Figure 2. X-ray scene is carried out with the range of angle from 10° to 80° with a constant speed of $2°\cdot min^{-1}$. The d values of the phases are matched with the standard JCPDS d values. [JCPDS – 85-0987, 84-1435, 86-1630, 86-1410].

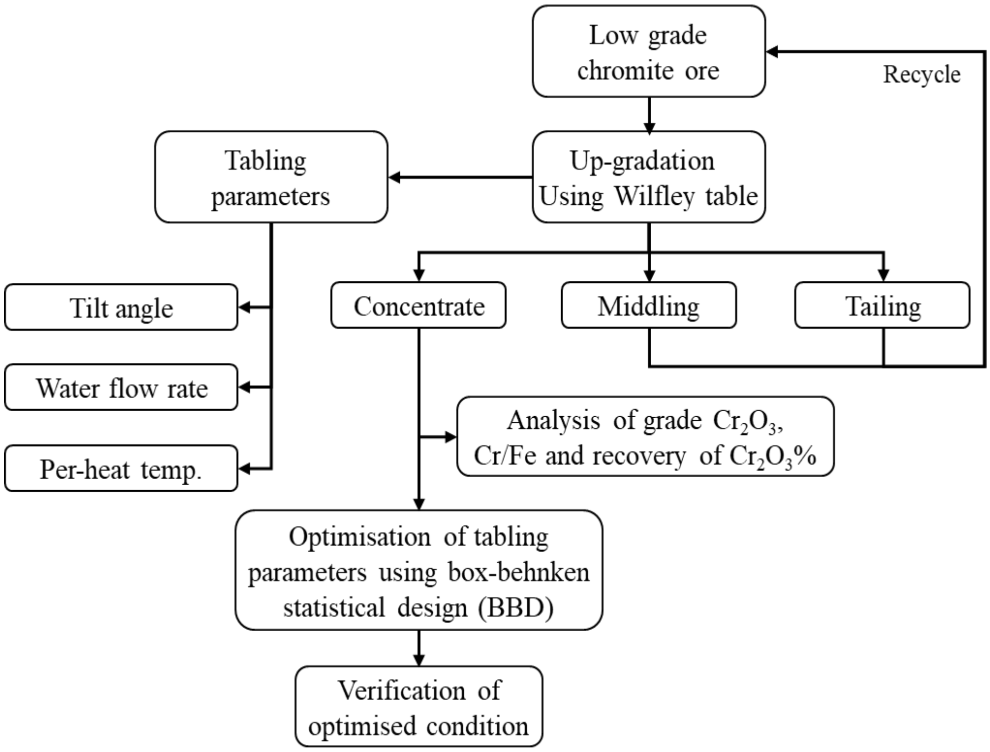

Figure 1. Schematic representation of experimental setup.

Table 1. WD-XRF analysis report of low grade chromite ore.

Cr_2O_3	Fe	Mn	Al_2O_3	SiO_2	TiO_2	K_2O	Ni	P	S	V_2O_5	CaO
26.88	23.72	0.55	21.07	0.92	0.29	0.004	0.1	0.02	ND	0.172	0.02

Table 2. EDX result for the low grade chromite ore.

Element	Weight %	Atomic% (α)
O K	36.89	57.43
Al K	12.65	13.95
Si K	2.23	2.70
Cr K	15.62	6.98
Fe k	28.64	14.01

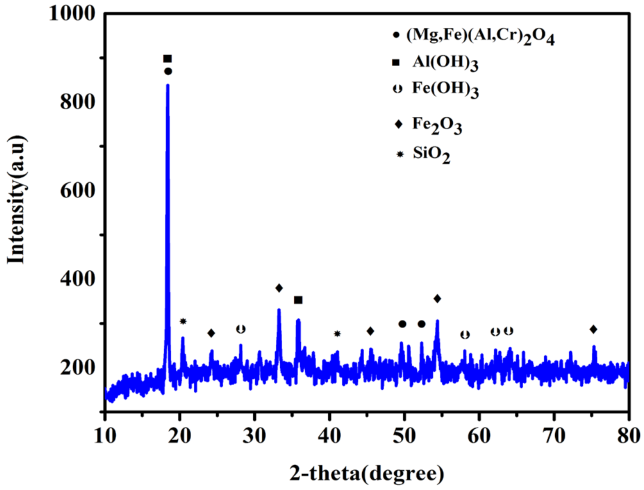

Figure 2. XRD analysis of raw grade chromite ore.

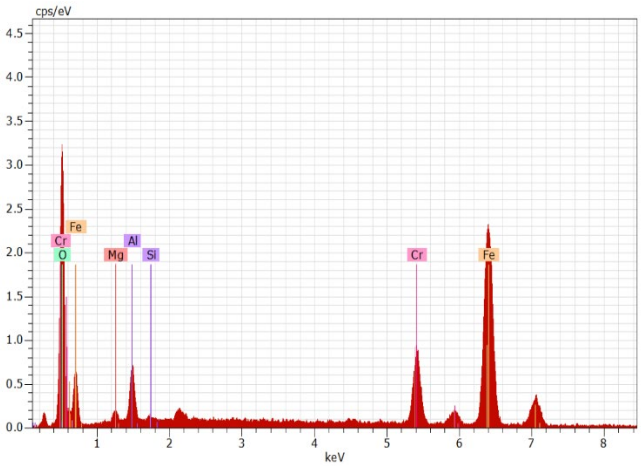

Figure 3. EDX spectrum of the element inside the chromite ore.

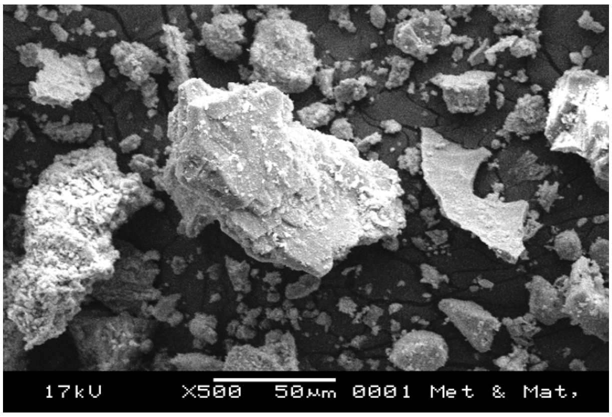

Figure 4. SEM image of low grade chromite ore.

2.2.3 SEM-EDX analysis of chromite ore

The elemental composition of raw chromite ore has been listed in Table 2 and EDX spectrum shown in Figure 3. The result indicated that Fe, Al, Cr and O are the major element present in the mineral. The α value ratio of Fe to Mg and Cr to Al is 2.84 and 0.50 respectively. The values indicated that the raw material is enriched with iron and aluminium. In the ore some of Cr is replace by Al which is also confirmed by XRD pattern (Figure 2). It is observed from Figure 4 the liberation size of the investigated chromite is 75 µm.

2.2.4 Distribution of elements

The brighter colour in Figure 5 indicated the presence of different elements. It is clearly observed that iron is widely distributed in ore. However distribution pattern of Al and Cr is quite similar. In the case of Mg and Si EDX mapping shows darker colour which indicates that the raw material contained lower amount of these elements.

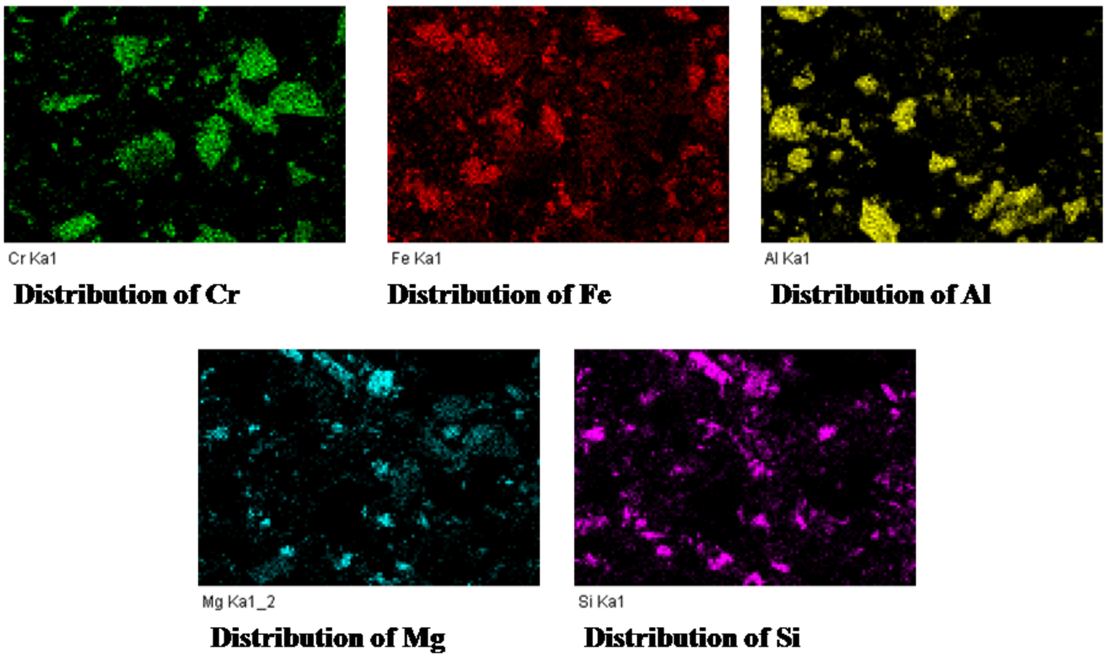

Figure 5. Elemental analysis of low grade chromite ore.

2.2.5 TG-DTA analysis of chromite

Thermal analysis represents the weight lose and heat flow with increasing temperature (0°C to 1000°C). The TG-DTA curve shows (Figure 6) that at 300°C their is sharp weight loss but after 800°C it is very marginal. Therefore 1000°C is chosen as the upper limit for pre-heat treatment.

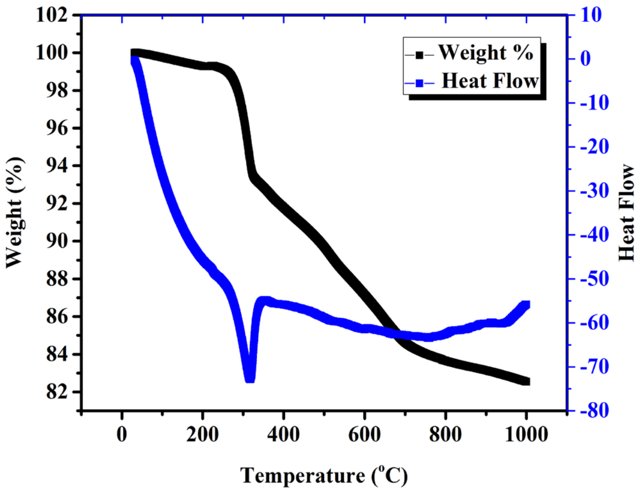

Figure 6. TG-DTA analysis of low grade chromite ore.

2.3 Pre-heat treatment of chromite ore

Composition of chromite ore obtained from earth crust is very complex in nature where main compound (chromium oxide) is physically and chemically combined with different types of gangue materials. Due to such complex mineralogy it is very difficult to separate gangue materials from chromite ore. It is observed that pre-heat treatment is effective in inducing thermal shock which might lead to change in the structure of ore mineralogy. Heating at a particular temperature followed by air cooling generates fissures in ore body which may help in separation of gangue from the ore body. In Figure 7, the pattern of cracks & fissure present in chromite ore particles is shown at different magnification of before and after heat treatment of chromite ore at different temperature. In Figure 7(a-c); the SEM image of raw chromite ore at 100x, 500x, and 1000x magnifications has been shown, respectively. Similarly Figure 7(d-f); the SEM images of pre-heated chromite ore at 514°C have been shown at different magnifications. The SEM images of pre-heated chromite ore at 1000°C have been shown at different magnifications as shown in Figure 7(g-i). It can be seen from the SEM images of the raw (after crushing) and pre-heat treated sample that cracks & fissures are predominantly present in the pre-heated sample whereas the raw sample (after crushing) have no such significant cracks & fissures. The selection of heating temperature is very crucial because at high temperature fusion of low melting phases might take place which in turn will increase the specific density of the sample.

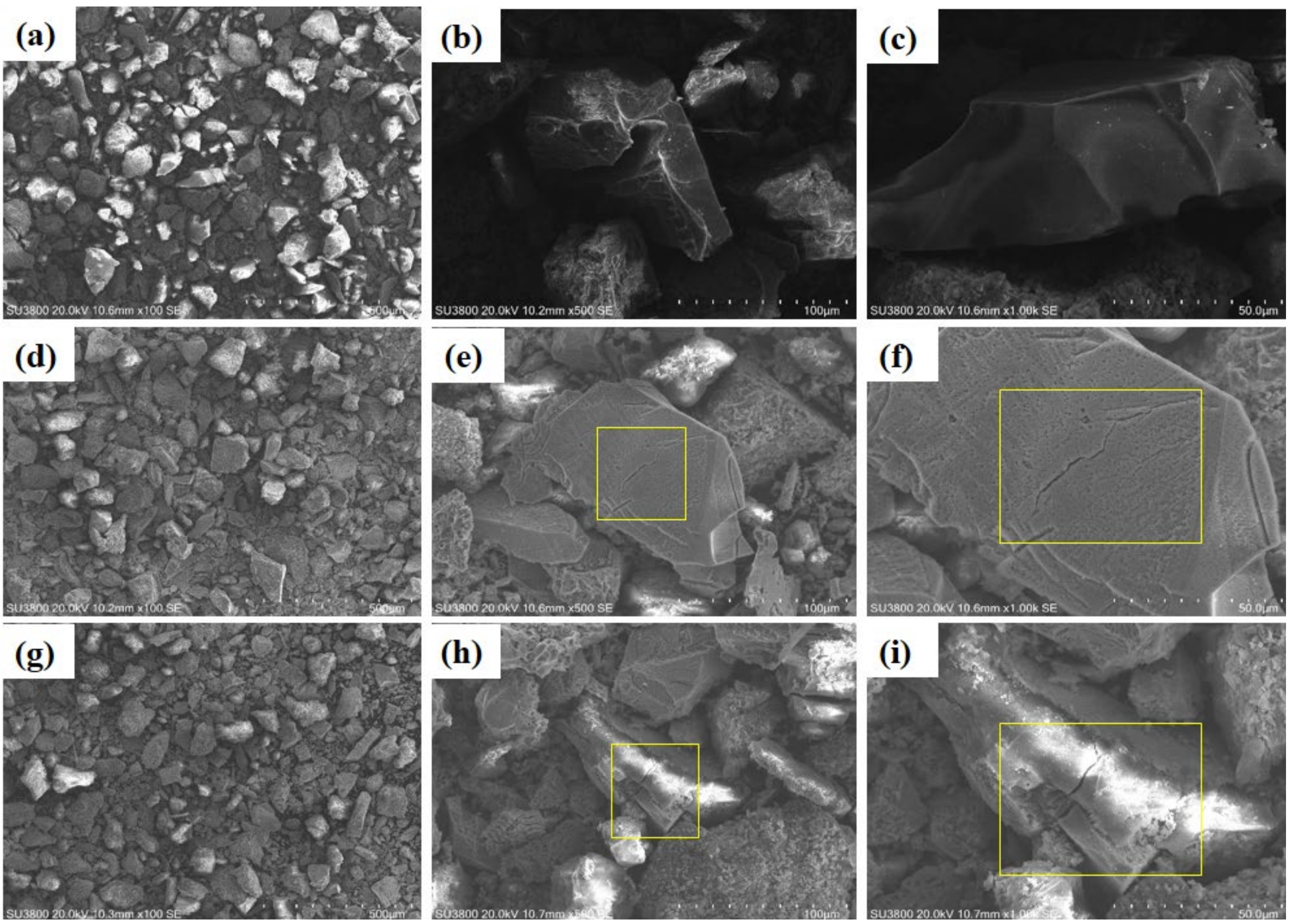

Figure 7. SEM image of chromite ore fines at different magnification of before (a,b,c) and after heat treatment at 514°C (d,e,f) and 1000°C (g,h,i).

In this investigation the pre-heat treatment of low grade chromite ore has been carried out under three conditions of temperature i.e. at room temperature (28°C), 514°C and 1000°C in a raising hearth furnace. Samples are then subjected to tabling maintaining all the other parameters (tilt angle and water flow) as suggested by BBD model for up gradation.

2.4 Application of the box–behnken design

For minimizing the number of experiments box-behnken statistical design has been selected with 3 factors, 3 levels which gives a set of 17 runs for the optimization study. The set of experiments (N) provided by BBD is calculated by the equation; $N = 2K (K-1) + C$, where K represents the number of factors and C is the number of central points. The Design Expert 8.0.7.1 software has been used for this purpose.

In this work, tabling of chromite ore has been carried out to understand the important effect of the input parameters on the response i.e. Cr/Fe ratio, grade (%) and recovery (%) of Cr_2O_3 in the concentrate fraction. The response is generated in form of a second-order polynomial equation with the interaction among the influencing factors which is given in following equation:

$$X\ (response) = Y + a_1A + a_2B + a_3C + a_4AB + a_5AC + a_6BC + a_7A^2 + a_8B^2 + a_9C^2$$

[Y is constant, a_i is regression coefficient, and A, B, C are independent variables [24-29].

The Table 3 represents the input parameters where each factor has three levels. Using BBD three factors-three levels experiments have been carried out. The optimization of process parameters have been done by examining 17 set of experiments instead of 27. With the help of mathematical modeling, relation between the parameters and the response has been established. The interaction effects of input parameters on the response have been shown in response surface plots. The statistical and correlation analysis of different input parameters on the response have been performed by the analysis of variance (ANOVA).

3. Results and discussion

3.1 Statistical analysis

Maintaining all the conditions given by box–behnken design, 17 set of experiment have been done in laboratory scale wilfley table. Cr:Fe, percentage Cr_2O_3 present in concentrate (grade Cr_2O_3%) and percentage Cr_2O_3 recovery have been calculated for each experiment which is shown in Table 4. It is observed that maximum Cr_2O_3 found in concentrate is 48.50% with the recovery of 38.42% and 39.62%. On the other hand maximum Cr_2O_3 recovery percentage is 70.08% with grade of Cr_2O_3 being 44.33%. Beside this maximum Cr:Fe ration is found to be 2.17 with recovery of 38.42%. The grade (g) and recovery (r) of Cr_2O_3 is clearly found to be increased when tabling is carried out by pre-heat treated chromite ore keeping all other experimental parameters same. It is observed that at 2° tilt angle, water flow rate 6 l/min, the grade and recovery of Cr_2O_3% at room temperature and at 1000°C are 40.90 (g), 63.99 (r) and 46.09 (g), 68.00 (r); respectively. So, tabling of pre-heat treated samples under the same conditions have shown better grade and recovery.

3.2 Analysis of variance (ANOVA)

The ANOVA of the quadratic model for Cr/Fe ratio, grade Cr_2O_3% and Cr_2O_3 recovery percentage is shown in Table 5. The model F-values for Cr/Fe ratio, grade Cr_2O_3% and Cr_2O_3 recovery % are 20.10, 7.89, 21.60 respectively, which implies the model is adequate. There is only a 0.63% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.05 indicate model terms are significant. The coefficient of variation (C.V.) of 2.44%, 2.18% and 5.31% indicates the dependability of experiment is good. R-squared above 0.90 implies that the models are well in agreement with the experimental data. R^2 values for Cr/Fe grade Cr_2O_3% and Cr_2O_3 recovery % are 0.9627, 0.9103 and 0.9652 respectively.

The relationship between grade of Cr_2O_3 (%) and Cr_2O_3 Recovery % with the input parameters as genarated by BBD has been shown in terms of second order polynomial equation as given below.

Cr:Fe = + 0.98077 + 0.032841* tilt angle + 0.086556 * water flow rate + 7.43588E-004 * pre-heat temp + 3.75000E-003 * tilt angle * water flow rate - 2.74348e^{-005} * tilt angle * pre-heat temp - 5.91564e^{-005} * water flow rate * pre-heat temp + 3.33333e^{-003} * tilt angle^2 - 3.12500e^{-003} * water flow rate^2 - 1.05844e^{-007} * pre-heat temp^2.

Grade Cr_2O_3 = + 32.67748 - 0.52525 * angle + 2.28609 * water flow rate + 5.16655e^{-003} * pre-heat temp. + 0.000000 * angle * water flow rate - 7.47599e^{-004} * angle * pre-heat temp. -2.31481e^{-005} * water flow rate * pre-heat temp. + 0.18436 * angle^2 - 0.14581 * water flow rate ^{2} - 1.37598e^{-008} * pre-heat temp.^{2}

Cr_2O_3 recovery = + 85.83743 - 0.43742 * angle- 8.36900 * water flow rate + 0.026752 * pre-heat temp. - 1.31042 * angle * water flow rate - 1.09568e^{-003} * angle * pre-heat temp. + 7.66461e^{-004} * water flow rate * pre-heat temp. + 0.57356 * angle^2 + 0.94238 * water flow rate^2 - 1.69584e^{-005} * pre-heat temp.^{2}

Table 3. Assignment of the levels to the factors.

Levels	Levels	Levels	
		Lower	Upper
A: Tilt angle	°	2	8
B: Water flow rate	l/min	4	8
C:Quenching temperature	°C	28	1000

Table 4. Box-behnken experimental designs.

Sl. No.	Control factors			Experimental results		
	Tilt angle (°)	Water flow rate ($L \cdot min^{-1}$)	Pre-heat temp. (°C)	Cr:Fe	Grade Cr_2O_3 (%)	Total Cr_2O_3 recovery (%)
1	5	6	514	1.88	44.15	52.38
2	2	4	514	1.67	41.92	68.21
3	5	8	28	1.89	44.71	47.36
4	8	6	28	2.09	47.67	42.00
5	2	6	1000	1.76	46.09	68.00
6	5	8	1000	1.82	44.12	46.44
7	8	4	514	1.98	46.09	68.00
8	5	6	514	1.89	44.10	53.22
9	8	8	514	2.17	48.50	38.42
10	5	6	514	1.87	44.13	51.23
11	5	4	28	1.75	42.93	59.07
12	8	6	1000	2.13	48.50	39.62
13	5	6	514	1.88	44.13	51.21
14	5	4	1000	1.91	42.43	55.17
15	2	8	514	1.77	44.33	70.08
16	5	6	514	1.88	44.19	51.20
17	2	6	28	1.56	40.9	63.99

Table 5. Statistical results of the ANOVA.

Statistical results	Response		
	Cr/Fe	Grade Cr_2O_3 %	Cr_2O_3 recovery %
Model F-value	20.10	7.89	21.60
Model prob>F	0.0003	0.006	0.0003
C.V. %	2.44	2.18	5.31
R-squared	0.9627	0.9103	0.9652
R^2-adjusted	0.9131	0.7949	0.9219
Sum of square	0.38	67.40	1629.51
Degree of freedom	9	9	9
Standard deviation	0.046	0.97	2.90

3.3 Verification test

Figure 8(a-c) shows the plot of predicted versus actual Cr/Fe ratio, grade (%) of Cr_2O_3 in the concentrate and Cr_2O_3 recovery percentage; respectively. Experimental data points present in the figures are very close to the predicted data points provided by BBD. The figures clearly show that there are no such serious violations of the assumptions that underlie the analysis [30]. The errors are distributed in a very significant manner, which is marginal. Therefore the results predicted by the BBD are in good agreement with the experimental results.

Response surface polts give better understanding of interactions between two independant variables and the other variable is located at center level. From the Figure 9(a-b) it is observed that with increase in water flow rate and tilt angle, the Cr/Fe ratio and grade Cr_2O_3 in concentrate fraction increased but the Cr_2O_3 recovery decreased as shown in Figure 9(c). The gungue meterials are pushed up by water when the tilt angle and water flow rate levels are high hence, gungue meterials are increased in tailing fraction, resulting in higher Cr_2O_3% in concentrate fraction. As weight percentage decreases in concentrate fraction at higher level of tilt angle and water flow rate, percentage Cr_2O_3 recovery is decreased in concentrate fraction.

The effect of water flow rate and pre-heat treatment of chromite ore on Cr/Fe ratio, grade (%) of Cr_2O_3 in the concentrate & Cr_2O_3 recovery percentage is shown in Figure 10(a-c) respectively. Maximun Cr_2O_3 recovery in concentrate fraction is found at intermidiate level of pre-heat treatment of chromite ore and water flow rate. Pre-heat treatment also increase the Cr/Fe and grade Cr_2O_3 in concentrate fraction. This happens due to the thermal shock induced by the pre-heat treatment followed by air cooling which leads to change in the structure of ore mineralogy which is responsible for the good separation of gangue materials from chromite ore.

Similarly Figure 11(a-c) represent the effect of tilt angle and pre-heat treatment of chromite ore on Cr/Fe ratio, grade (%) of Cr_2O_3 in the concentrate & Cr_2O_3 recovery percentage; respectively. Maximum Cr/Fe ratio is found to be at highest level of tilt angle and intermediate level of pre-heat tempareture but in case of grade Cr_2O_3 percetage it is maximum at highest level of tilt angle and lowest level of pre-heat tempareture. But Cr_2O_3 recovery is maximum in intermidiate levels.

From the above discussions it can clearly concluded that quite similar effects of input parameters is shown for the Cr/Fe ratio and grade Cr_2O_3% in concentrate fraction. But opposite results are observed for Cr_2O_3 recovery percentage. It is because recovery percentage depends upon the weight percentage of concentrate fraction along with Cr_2O_3%. The corresponding values of input parameters for optimizing each response is given by BBD model, which is shown in Table 6. The result is generated by the model according to desirability of the response. Predicted value of responses are also reflected the relation between Cr/Fe ratio, grade Cr_2O_3% and Cr_2O_3 recovery percentage. The best result is obtained in terms of highest desirability at 8° tilt angle, 4l/min water flow rate and per-heat of chromite ore at 443.72°C. The chromite sample is subjected to wilfley table maintaining the above predicted input parameters for verification of the predicted response. The experimental results are matching reasonably to the predicted results which is given in Table 6.

The XRD analysis report of chromite ore concentrate after tabling is shown in Figure 12. The major peak is found to be aluminian chromite $Fe(Al,Cr)_2O_4$ along with hematite and aluminium oxide. It is clearly observed that the intensity of the $Fe(Al,Cr)_2O_4$ peak is much higher in chromite concentrate compare to raw chromite ore indicating higher grade of concentrates.

It is observed from Table 7 that the weight percentage of chromium has significantly increased in the concentrate fraction. The EDX spectrum is shown in Figure 13, which reflects that the peak intensity of chromium is highest among all elements present in the chromite ore. The α value ratio of Cr to Al in raw chromite ore and chromite concentrate is 0.50 and 2.0, respectively. The values indicate that the raw material is enriched with aluminium whereas chromite concentrate is enriched with chromium.

The distribution of different elements present in the chromite concentrate is shown in Figure 14. From the EDX elemental mapping, it can be seen that chromium is distributed homogeneously along with iron in the crystal grain. The concentration of silicon and aluminium in concentrate fraction is lower than the raw chromite ore.

The SEM image of the beneficiated chromite concentrate at different magnifications has been given in Figure 15(a-b). Cracks & fissures are clearly present in the crystal grain. Pre-heat treatment changes the surface morphology of the upgraded material which may inturn increase the reducibility.

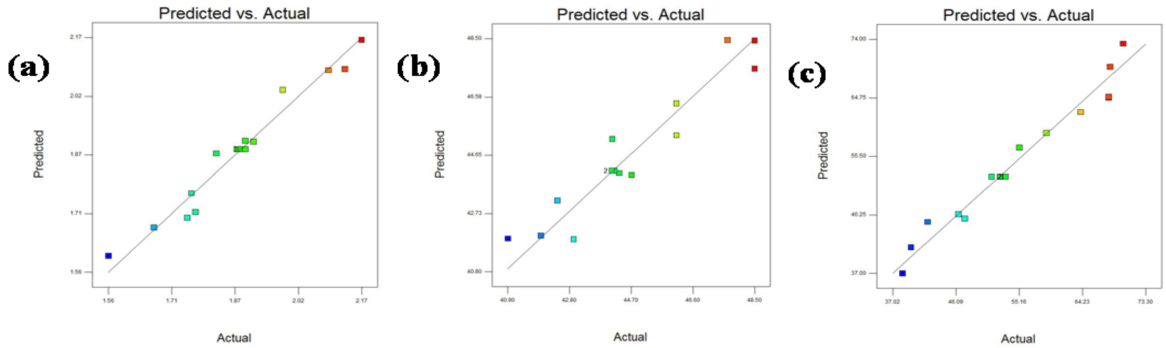

Figure 8. Actual versus predicted plots for (a) Cr/Fe ratio (b) grade (%) of Cr_2O_3 in the concentrate (c) Cr_2O_3 recovery percentage.

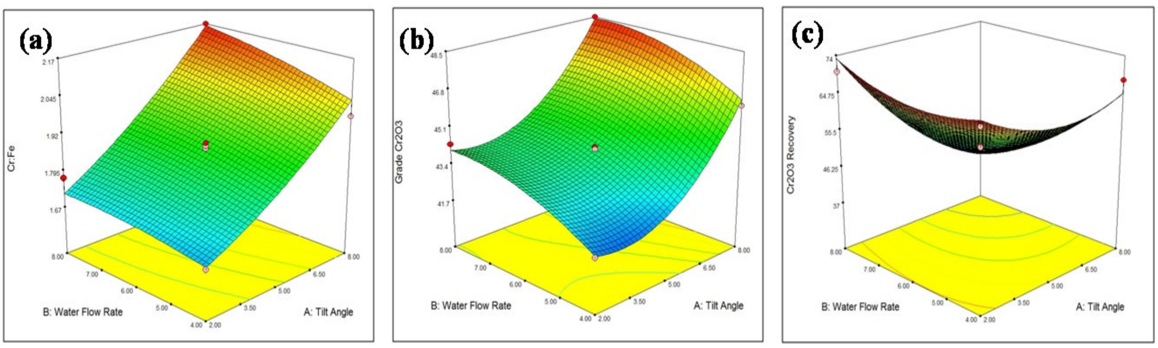

Figure 9. Response surface plots showing effect of tilt angle vs water flow rate on (a) Cr/Fe ratio (b) grade (%) of Cr_2O_3 in the concentrate (c) Cr_2O_3 recovery percentage.

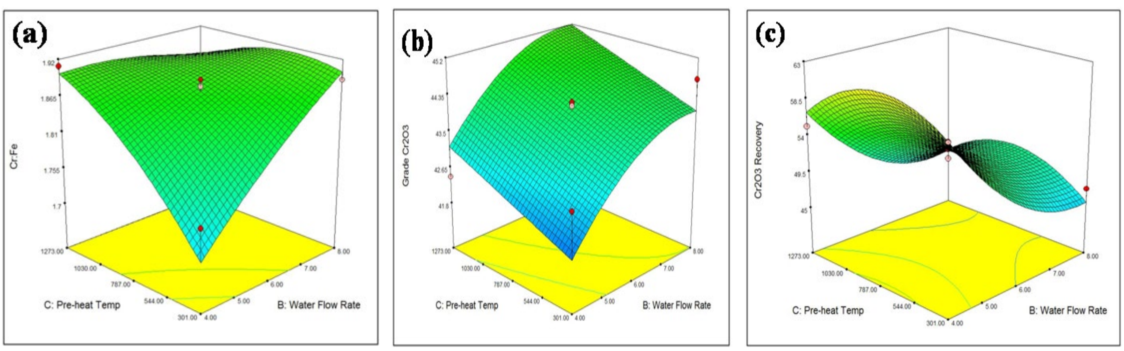

Figure 10. Response surface plots showing effect of pre-heat temperature vs water flow rate on (a) Cr/Fe ratio (b) grade (%) of Cr_2O_3 in the concentrate (c) Cr_2O_3 recovery percentage.

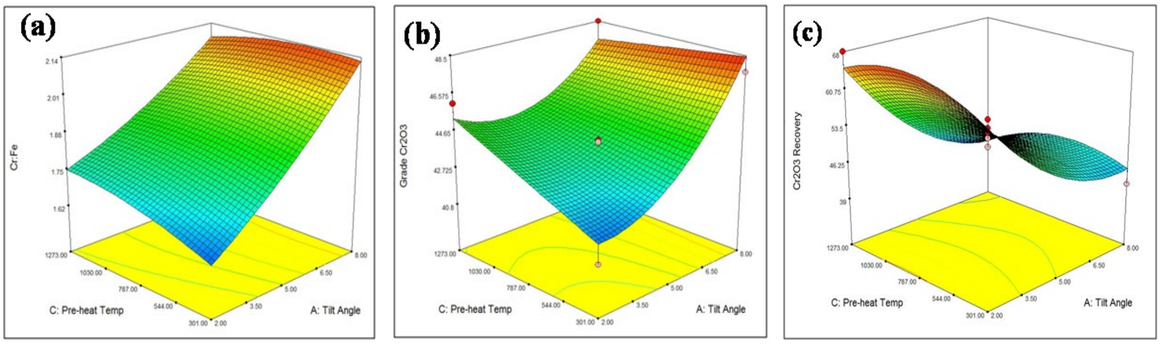

Figure 11. Response surface plots showing effect of pre-heat temperature vs tilt angle rate on (a) Cr/Fe ratio (b) grade (%) of Cr_2O_3 in the concentrate (c) Cr_2O_3 recovery percentage.

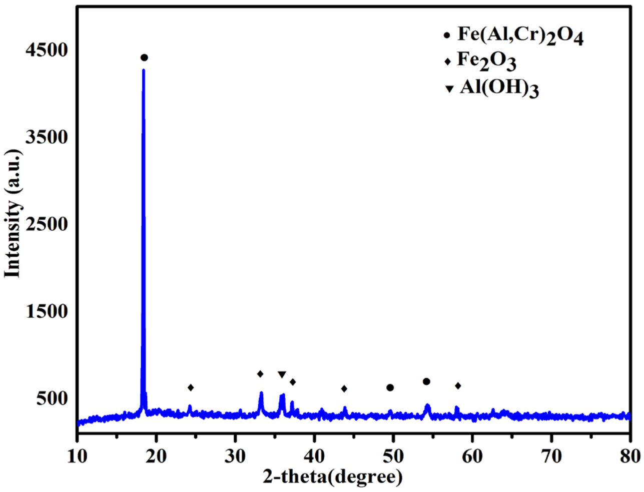

Figure 12. XRD analysis of chromite ore concentrate.

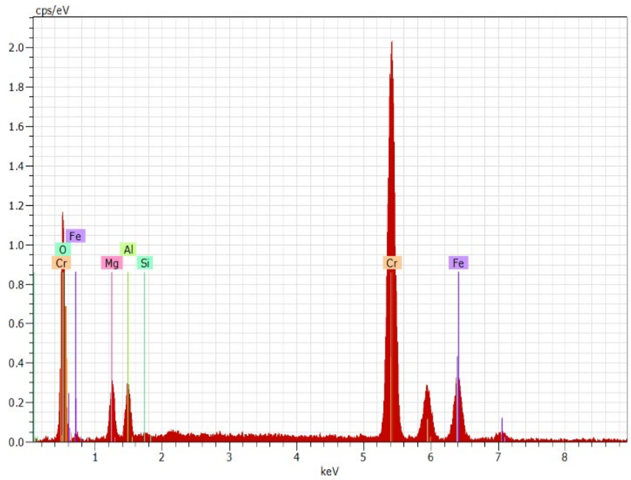

Figure 13. EDX spectrum analysis of the element inside the chromite concentrate.

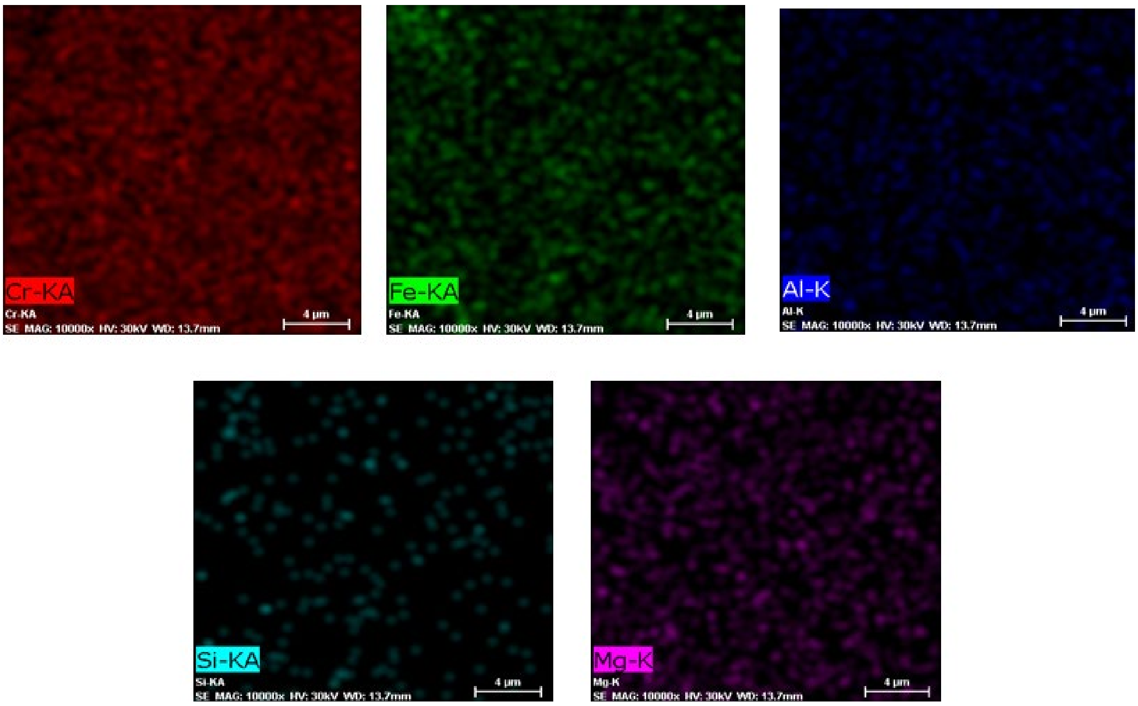

Figure 14. Elemental analysis of low grade chromite ore.

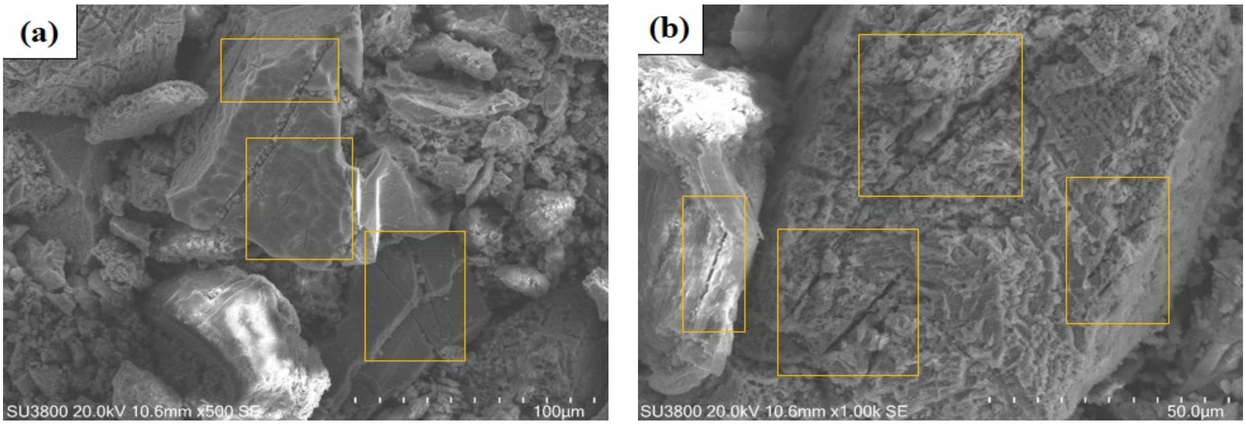

Figure 15. SEM image of chromite ore concentrate.

Table 6. Predicted and experimental results of chromite concentrate at predicted input parameters given by BBD.

Sl. No.	Control factors			Experimental results		
	Tilt angle (°)	Water flow rate ($L\cdot min^{-1}$)	Pre-heat temp. (°C)	Cr:Fe	Grade Cr_2O_3 (%)	Cr_2O_3 recovery (%)
Predicted	8.00	4.00	443.72	2.02	46.43	65.09
Experiment	8.00	4.00	443.72	1.91	46.02	65.95

Table 7. EDX result of chromite concentrate .

Element	Weight%	Atomic% (α)
O K	32.97	57.27
Al K	7.81	8.38
Si K	1.42	1.94
Cr K	35.96	16.75
Fe K	16.83	8.60
Mg K	5.01	7.06

4. Conclusions

The mineralogical investigation shows that the tested sample is low grade chromite ore and ferruginous in nature. Microscopic analysis of chromite ore shows that the liberation size range of the chromite particle is 75 µm to 100 µm. X-ray diffraction study indicates that the structure of chromite ore is spinel in nature. The major phase present in the sample is berezovskite [$(Mg,Fe)(Al,Cr)_2O_4$]. The distribution of Al throughout the cross-section of chromite particle is higher than any other element present in the sample. The gravity separation by the means of wilfley table (lab scale) is found to be very effective for upgradation. Present work represents the optimization of tabling process parameters with the help of BBD model. Amongst all the input parameters, tilt angle is the dominant parameter as compared to the other two parameters. Pre-heat treatment has a very significant effect on the Cr_2O_3 recovery percentage due to generation of cracks & fissures in the ore bodies during air quenching. Per-heat treatment at particular temperature helps the separation of gangue minerals from chromite ore. The experiment R^2 values for Cr/Fe ratio, grade of Cr_2O_3 and Cr_2O_3 recovery are found to be 0.96, 0.91 and 0.96 respectively which shows the experimental results are in good agreement with the predicted values as given by BBD model. The ANOVA results also depict the same observation for different values in statistical model. The 3D response surface plots show the relation between the different input parameters with the response. The maximum Cr/Fe ratio, grade Cr_2O_3% and Cr_2O_3 recovery percentage achieved in present experiment are 2.17, 48.50 and 70.08; respectively under different conditions keeping the response maximum. It has been found that low grade chromite ore can possibly be upgraded upto 46.02% grade Cr_2O_3, containing a Cr/Fe ratio of 1.91 with recovery rate of 65.95% in concentrate fraction under the following conditions: tilt angle (8°), water flow rate 4 $L\cdot min^{-1}$ and pre-heat temperature of 443.72°C, suggested by BBD model. The study of extent of reduction of the upgraded raw materials after being pre-heat treated can give a better understanding of the impact of pre-heat treatment on the reducibility of the upgraded material. If positive results can be achieved, scaling up of the test, for commercialization can be carried out in the future.

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